Trace Organics Data Anomaly Form

Date(s) Occurred: *10-05-2006, 10-20-2006*

WG #(s): *88422, 88423*

☐ **All samples in WKGP(s) or Sample #(s):** *L40468-(1, 4, 5)*

Project #(s): *423589-090-1*

Matrix: ☒ **Liquid** ☐ **Solid** ☐ **Air** ☐ **Tissue** ☐ **Calibration** ☐ **Other:**

I. Analysis/Extraction

☐ BNA	☐ BNALL	☐ EDC	☐ EDC-LVI
☐ CLPESTPCB	☐ PEST	☐ PCB	☐ OPPEST
☐ VOA-GCMS	☐ NWTPH-GX	☐ NWTPH-DX	☐ NWTPH-HCID
☐ BUTYL TIN	☐ AIRTOX	☐ AIR-SULFUR	

☒ Other: *PHPAHSOURCE, PCBLL*

☐ Subcontracted:

II. Instrument

GC/ICP/MS: ☐ P

GC/MS: ☐ D ☐ E ☐ J ☐ K ☐ L ☐ M ☒ N

GC: ☐ F ECD ☐ G ECD ☐ H FID ☐ H OI4450PID/FID ☐ I FID

Extraction/Cleanup: ☐ PFE ☐ GPC ☐ Other:

III. Type of Sample/Analytical Anomaly

☐ Values Outside of Control Limits:

1 ☐ Blank Contamination
2 ☐ SB/SBD Spike Recoveries
3 ☐ MS/MSD Spike Recoveries
4 ☐ LCS/SRM Recoveries
5 ☐ Initial Calibration
6 ☐ Performance Checks
7 ☐ ISTD % Differences
8 ☐ Surrogate Spike Recoveries
9 ☐ SB/SBD RPD
10 ☐ MS/MSD RPD
11 ☐ Sample/LD RPD
12 ☐ Continuing Calibration Checks
13 ☐ Tuning Criteria

14 ☐ Holding time exceeded by:
15 ☐ Insufficient sample amount.
16 ☐ Inappropriate storage, container or preservation.
17 ☒ Other

Anomaly Description: *(1) For sample L40468-1, the sample clogged the Speedisk. Problem appeared to be a high concentration of suspended algae in the sample. The flow was reduced so much that the last 500 ml of the sample had to be discarded. Unfortunately, the surrogate spikes (DMC surrogates, PCB field and in-house surrogates and BNA-LVI surrogates) were added prior to discarding the*

residual 500 ml volume. Therefore, we will have to adjust the surrogate level based on fraction of total volume lost and making the assumption that the sample was homogeneous before and at time of discard.

(2) For sample L40468-1, a significant portion of the sample split for PHPAHSOURCE analysis was spilled during preparation of dilutions. Estimated loss was 50% of the PHPAHSOURCE fraction.

(3) For sample L40468-5, the sample clogged the solid phase extraction disk while being poured through the Speedisk. Sample remaining in the Speedisk funnel assembly was returned to the original carboy. The volume of sample remaining in the carboy was then marked so that the actual volume passed through the Speedisk could be determined. Since there were no available Speedisks remaining to process the remaining volume separately, the sample was refirgerated overnight. The remaining sample was processed the following day using newly arrived Speedisks with separate lot number. Therefore, the two volume fractions were processed through the Speedisks on separate days. The separate sample fractions were subsequently processed through Na2SO4 drying and K-D concentration. At that point, the two fractions were combined into a single volume and then blown down under nitrogen gas to final volume of 1.0 ml.

(4) There were differences observed in the physical appearance and behavior and sample L40468-4 and L40468-5. These differences are of major concern since the two samples were supposed to be duplicates taken from the same sampling site. As mentioned above, clogging was a problem for L40468-5. However, clogging was not a problem for L40468-4.

The total volumes observed for the two samples were in the same ballpark (2990 ml for L40468-4 and 3080 for L40468-5), however, while L40468-4 had more particulate than L40468-5, L40468-5 appeared to have more suspended particulate (algae) compared to L40468-4. The suspended particulate or algae appeared to be associated with clogging of the Speedisk.

In addition, the pH for L40468-4 was measured at 6 while L40468-5 had a pH of approximately 8. The elevated pH for L40468-5 was not observied until after the first fraction was processed. Before processing the second fraction of L40468-5, the pH was adjusted to 6 using approximately 1 ml conc HCl.

(5) For documentation only, all samples concentrated to 1.0 m final volume and then split 50:50 for analysis for PHPAHSOURCE and PCBLL. Fraction for PCBLL was subjected to alumina and H2SO4 sample cleanup procedures according to standard KCEL SOP. PCBLL fraction then concentrated to 0.5 ml following clean-up procedures.

IV. Type of Project Anomaly

- ☐ SAP/Work Plan specified MDLs not met.
- ☐ SAP/Work Plan specified QC frequency or QC type not met.
- ☐ SAP/Work Plan specified methodology not used.
- ☐ Sample exceeds regulatory and/or hazardous waste limits.
- ☐ Sample data results are unusual or inconsistent with expected results.
- ☐ Other

Anomaly Description:

V. Corrective Action Taken

☐ Sample(s) re-analyzed ☐ Sample(s) re-prepared and re-analyzed
☒ Sample(s) reported "AS IS"
☐ Data qualified with the following flags:
☐ Other

Corrective Action Description: *(1) For sample L40468-1, the total volume for sample was measured at 3700 ml. Following the disard of 500 ml after clogging, the effective volume was 3200 ml. The adjustment factor to determine effective surrogate levels for the DMC surrogates, PCB field and in-house surrogates and BNA-LVI surrogates, is then (3200/3700) * 100% = 86.5%. The surrogate levels for sample L40468-1 were adjusted accordingly prior to calculation of surrogate recoveries. No significant impact on data is anticipated.*

(2) For sample L40468-1, a significant portion of the sample split for PHPAHSOURCE analysis was spilled during preparation of dilutions. Estimated loss was 50% of the PHPAHSOURCE fraction. The sample concentrate was homogeneous at time of spillage and sufficient volume remained to prepare 1:10 dilution. Based on sample appearance and previous experience with similar samples, only the 1:10 dilution is likely to be analyzed so no significant impact on data is expected.

(3) For sample L40468-5, observed surrogate recoveries were consistent with those observed for other samples in the same workgroup. No significant quantitative impact on data is anticipated for sample L40468-5.

(4) Quantitative impact of observed differences between samples L40468-4 and L40468-5 is unknown.

(5) Documentation only. No quantitative impact on data.

VI. Potential Effects on Data Quality

Based upon the expected performance of this method:

☐ It is likely the observed anomaly influenced the reported value(s).
☒ It is unlikely the observed anomaly influenced the reported value(s).
☐ The observed anomaly may have influenced the reported value(s).
☐ It is unknown whether or not the observed anomaly affected the reported value(s).

Explanation:

	Signatures	Signature Dates
Reported By: *Jack Gudeman*		
Reviewer: *Mike Doubrava*	_____	_____
Supervisor: *Dana Walker*	_____	_____
QA Officer: *Colin Elliott*	_____	_____
(For QA1 only)	_____	_____
cc: LPM:		